**6.3**          **Technology for Real-Time Measurement**
**of Surface and Airborne Beryllium**

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# Abstract

The adverse health effects of exposure to airborne beryllium are well known. Personnel
exposure to beryllium during clean-up activities or during material processing is a major
health
risk within the DOE, DOD, and some private sector industries. Present laboratory-based
technologies for evaluating the concentration of airborne and surface beryllium do not
provide
the real-time analysis required to effectively protect workers.
In response to this need, Science & Engineering Associates, Inc. (SEA) is developing a
multi-function
beryllium monitor based on laser induced breakdown spectroscopy (LIBS). This system
will provide the capability to conduct continuous air monitoring for beryllium and the
analysis of
swipe or smear samples to detect beryllium contamination on surfaces and equipment.
The
instrumentation is expected to provide measurements of airborne beryllium to levels less
than 0.2
$\propto$g/m$_3$ , and measurements of surface contamination to less than 0.2 $\propto$ g/100 cm $_2$ for
swipe
samples. The instrument will operate in a fully automated mode for both the continuous
air
monitoring and swipe functions and the system will provide for an alarming function,
with the
trigger level set by the operator.
LIBS is an analytical technology that has been used for rapid, in-the-field measurements
for a
variety of analytes, with chromium, lead, and beryllium being some of the more common.
To

summarize, a pulsed, high-power infrared laser is focused to a small spot on the sample. The
very high energy density created at this surface rapidly heats the sample and the surrounding air,
creating plasma. Materials within the plasma are vaporized and the resulting atoms are electronically excited to emit light that is characteristic of the emitting elements. The intensity of
the light emitted by a particular element is proportional to the abundance or concentration of that
element in the mixture. Thus, by spectrally examining the emitted light, the abundance or concentration of a given element may be determined.

# Technology for Near Real-Time Measurement of Airborne and Surface Beryllium

**Steven Saggese, Ph.D. (PI)**
**Tom Kendrick (PM)**
**Science & Engineering Associates, Inc.**

**Presented by:**

**Bill Lowry**
**Science & Engineering Associates, Inc.**

# Outline

- Benefits of System

- Project Goals

- Scope of Work

- Technical Approach

- System Design

- Sample Data

- Schedule/Cost Status

# Benefits

- Provide rapid, in-the-field measurement results for airborne and surface beryllium contamination

- Lower risk of worker exposure to beryllium

- Fully automated operation in the CAM and wipe analysis modes, thereby reducing the labor costs associated with current air sampling schemes

# Project Goals

- Functional Performance
  - Provide a CAM function
  - Provide a wipe analysis function
  - Facilitate inclusion of spikes and blanks
  - Provide results traceable to recognized standards
  - Provide alarm capability to D&D personnel
- CAM Measurement Performance
  - Detection limit at or near 0.1 $\mu$g Be/m$^3$
  - OSHA peak of 5 $\mu$g Be/m$^3$ 15 minute cumulative average
  - Rocky Flats' action limit of 0.2 $\mu$g Be/m$^3$
- Wipe Measurement Performance
  - Detection limit of 0.2 $\mu$g Be/100 cm$^2$ or less

# Scope of Work

**WBS 1.0**
**Development of a Real-Time Measurement System for Airborne and Surfae Beryllium**

| WBS 1.1 Project Planning | WBS 1.2 Instrument Design | WBS 1.3 System Fabrication | WBS 1.4 Instrument Calibration | WBS 1.5 Field Testing | WBS 1.6 System Delivery |
|---|---|---|---|---|---|
| 1.1.1 — Management Plan, Cost Plan, NEPA | 1.2.1 — Requirements Analysis | 1.3.1 — Electronics Module | 1.4.1 — Calibration Test Plan | 1.5.1 — Develop Field Test Plan | 1.6.1 — Prepare Instrumentation Documentation |
| 1.1.2 — Kick-off Briefing | 1.2.2 — Methods Development | 1.3.2 — CAM/Swipe Module | 1.4.2 — Calibration & Validation Samples | 1.5.2 — Round One Field Testing | 1.6.2 — Prepare/Deliver ITSR |
| | 1.2.3 — Subsystem Hardware Design | 1.3.3 — Surface Module | 1.4.3 — Execute Calibration Test Plan | 1.5.3 — Design Refinements | 1.6.3 — Prepare/Conduct Final Briefing |
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# Technical Approach

- Filter-Based Sampling
- Laser-Induced Breakdown Spectroscopy (LIBS)
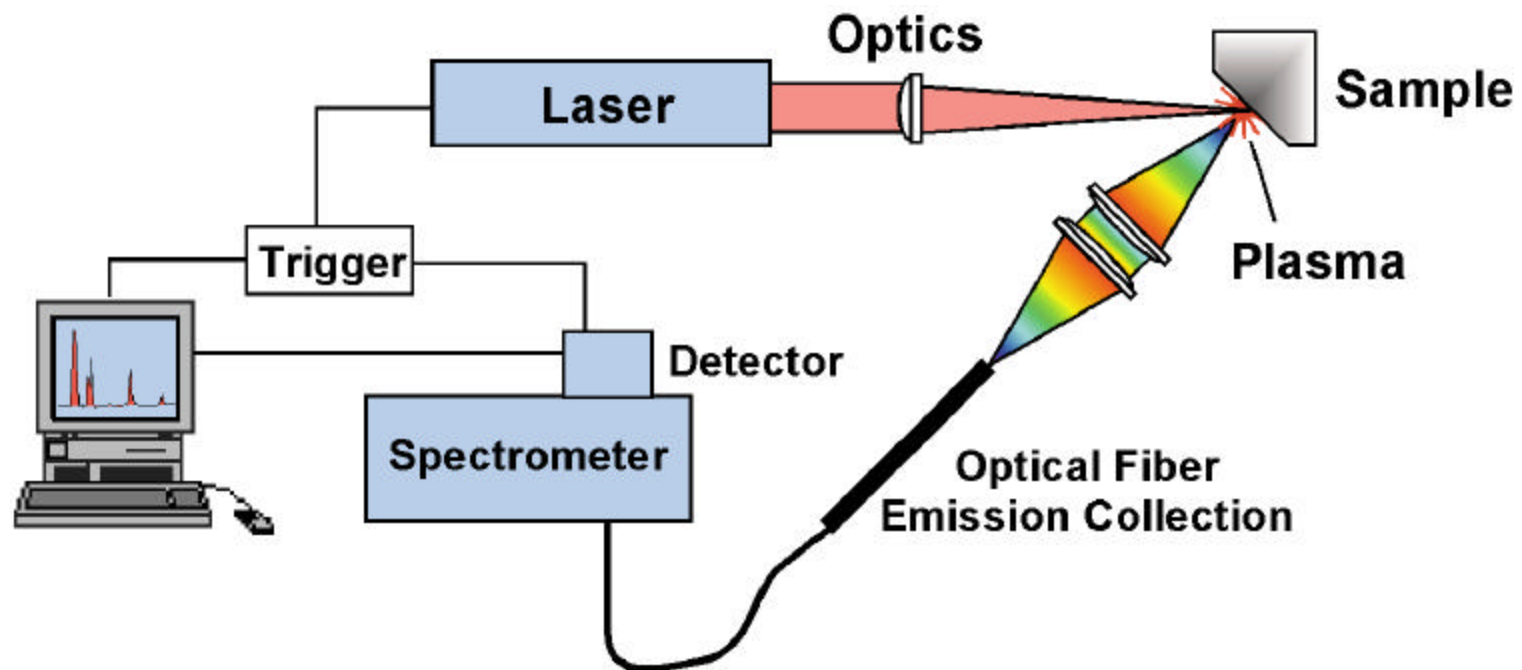- Multivariate Data Analysis

# Filter-Based Sampling

- Filter-based sampling consistent with standard air monitoring practices
- Filter-based sampling provides more material for evaluation when compared to direct analysis of air, resulting in:
  - Higher SNR
  - Lower Detection Limits
- Multiple modes of operation (CAM & Wipe)
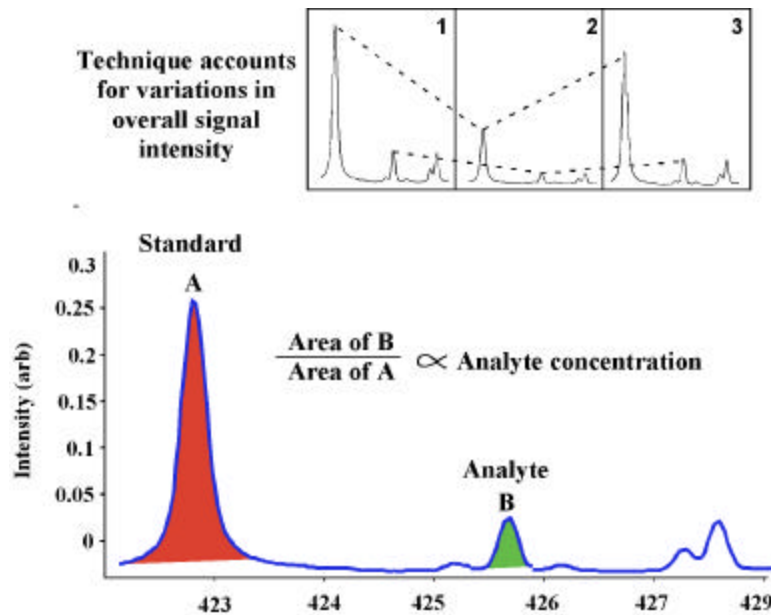- Allows for spikes and blanks

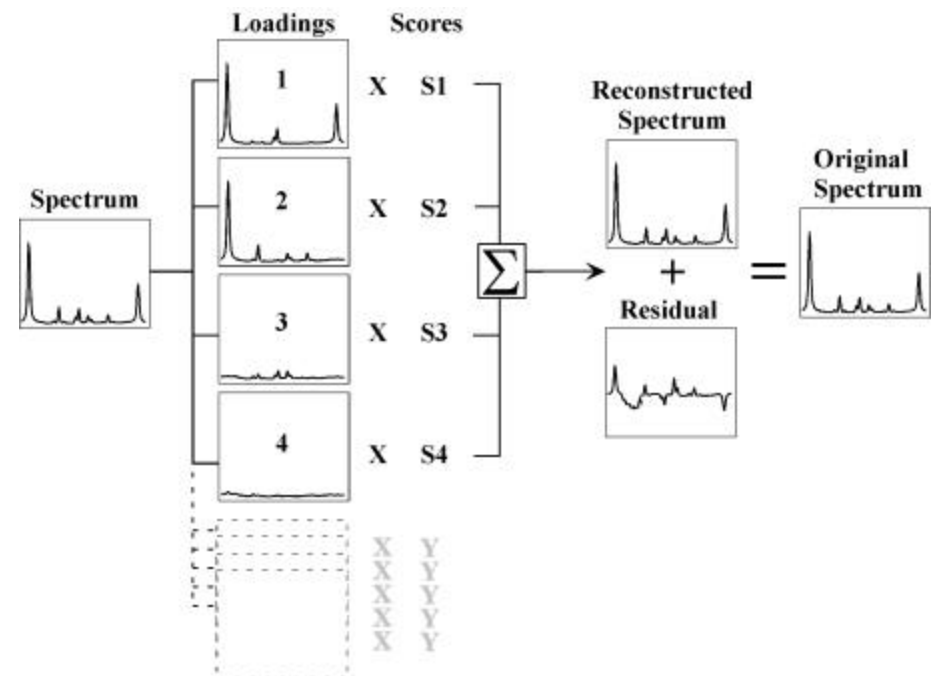# Laser Induced Breakdown Spectroscopy (LIBS)

**Ratio**

**Multivariate**
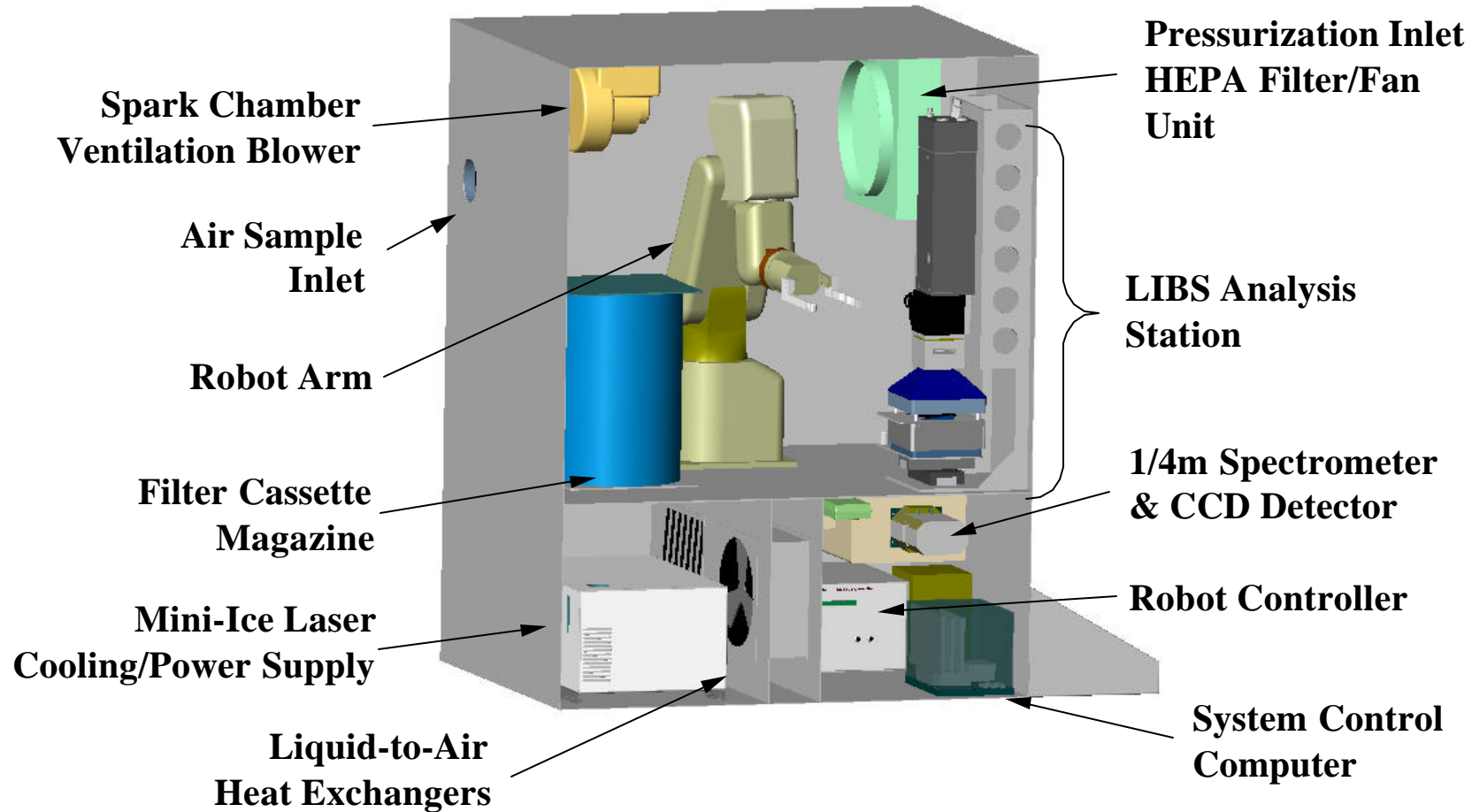
# Features of System Design

- Single unit for CAM and swipe analysis
- Windows-based GUI for system operation
- Fully automated operation in CAM and swipe modes
- CAM measurement update every 6 minutes
- 4-hour unattended operation in CAM mode (minimum)
- User-defined alarm levels
- Smooth exterior for easy decontamination
- Uses standard swipes and air filters

# System Design
## Overview

**Spark Chamber Ventilation Blower**

**Air Sample Inlet**

**Robot Arm**

**Filter Cassette Magazine**

**Mini-Ice Laser Cooling/Power Supply**

**Liquid-to-Air Heat Exchangers**

**Pressurization Inlet HEPA Filter/Fan Unit**

**LIBS Analysis Station**

**1/4m Spectrometer & CCD Detector**

**Robot Controller**
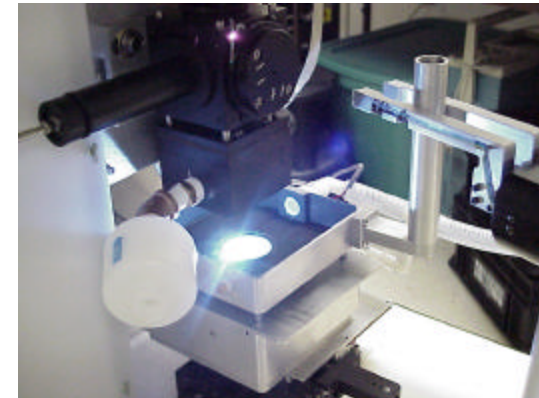
**System Control Computer**

# System Design
## LIBS Column
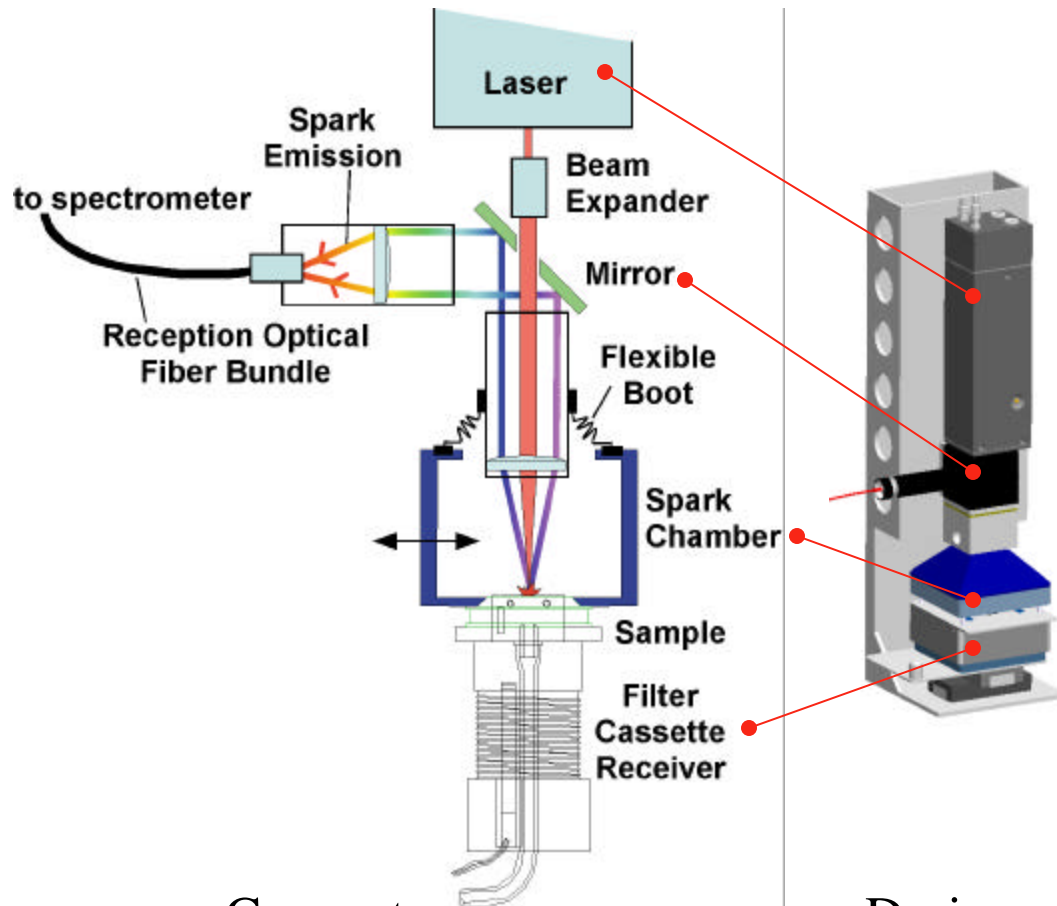


Concept ⟶ Design ⟶ Hardware

# System Design
## Filter Holder/ Spark Pattern



Disassembled
Filter Cassette

Scale in cm

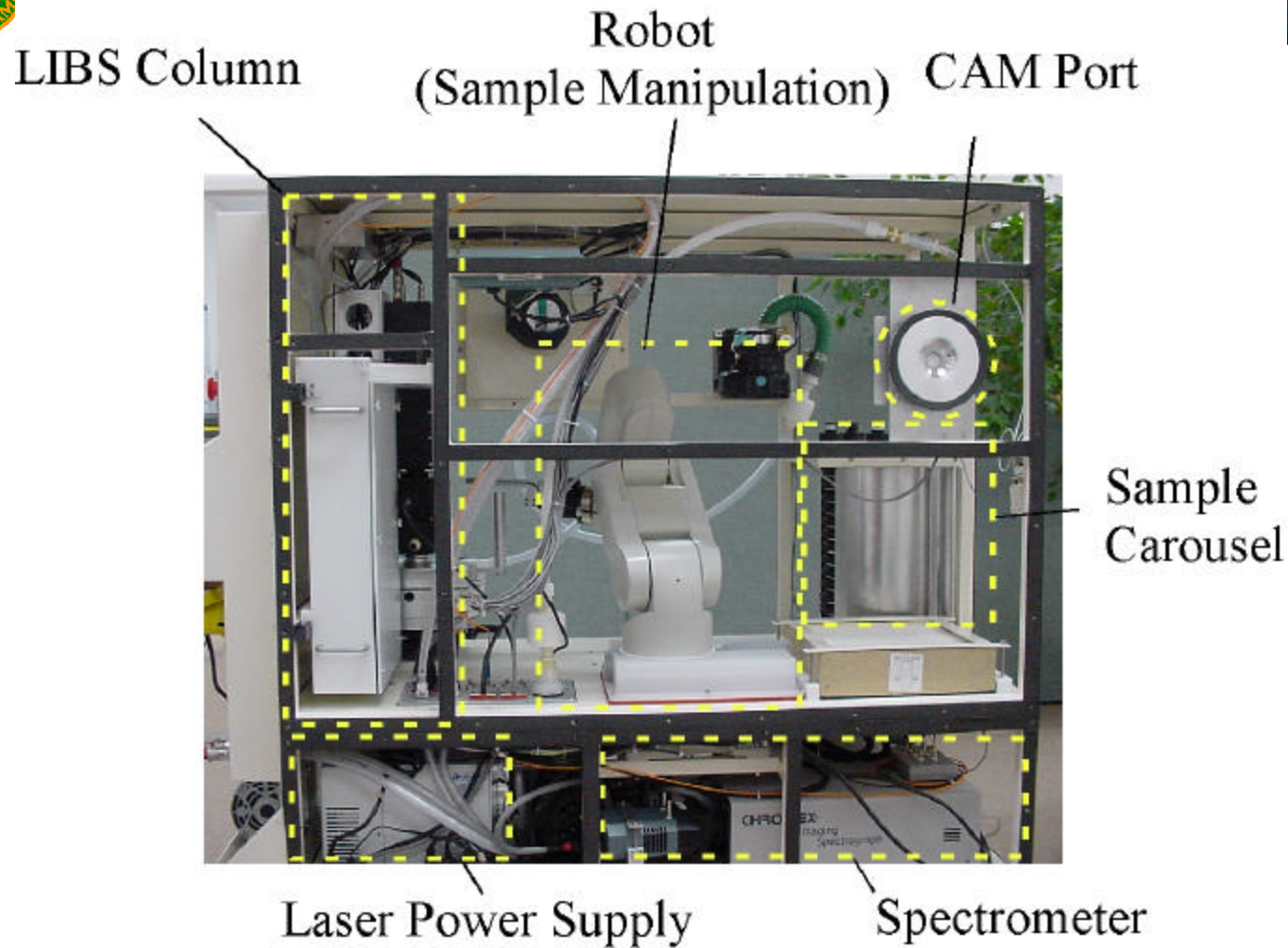0 1 2 3 4 5 10

Assembled
Filter Cassette
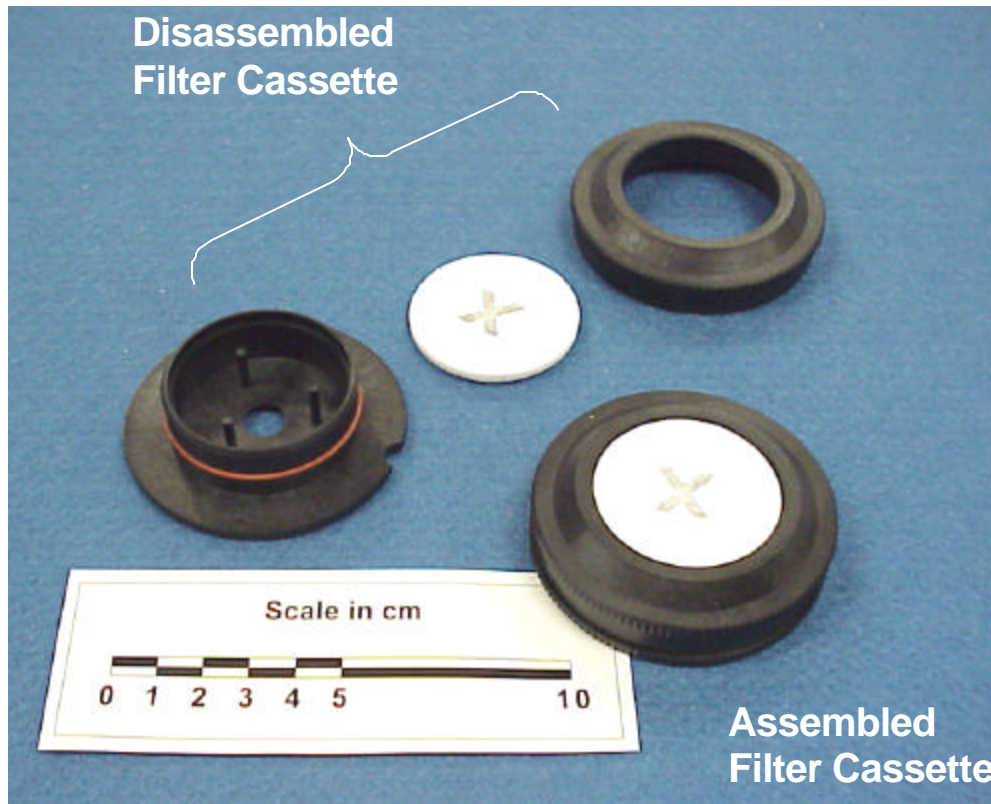


## Sparking Pattern
(on metal target)

# System Fabrication

# System Fabrication

## Close-up of Instrument Chamber



Laser Power Supply

Spectrometer

Heat Exchanger
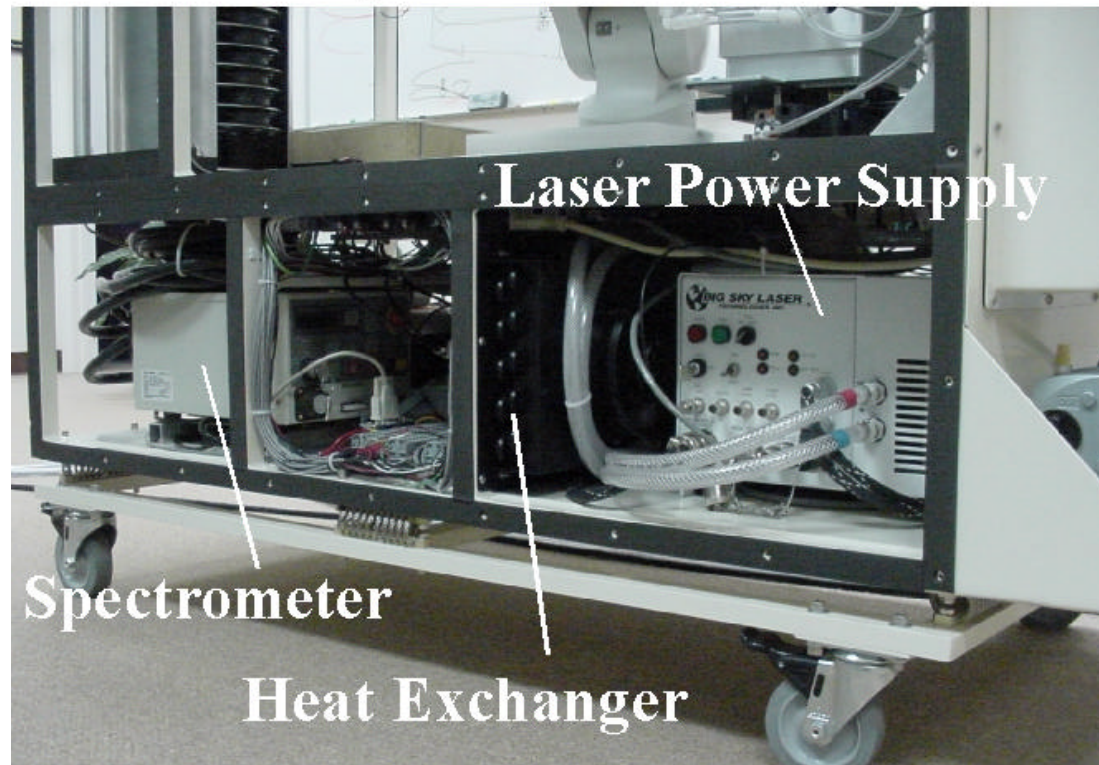
# System Fabrication, cont

**CAM Port**
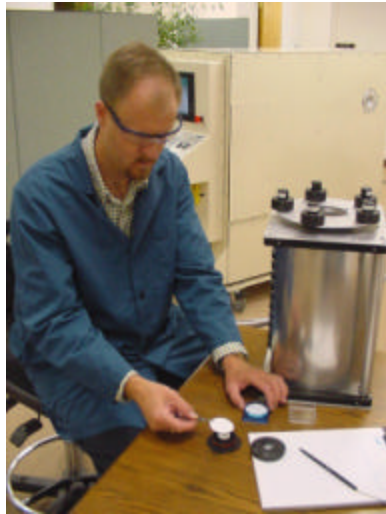


**Loading filter into holders**
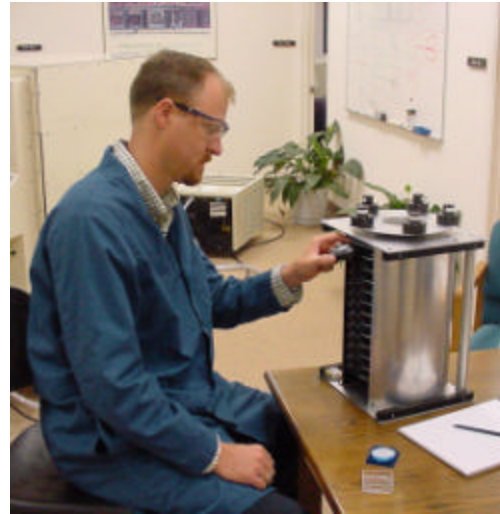
**Loading filter carousel**
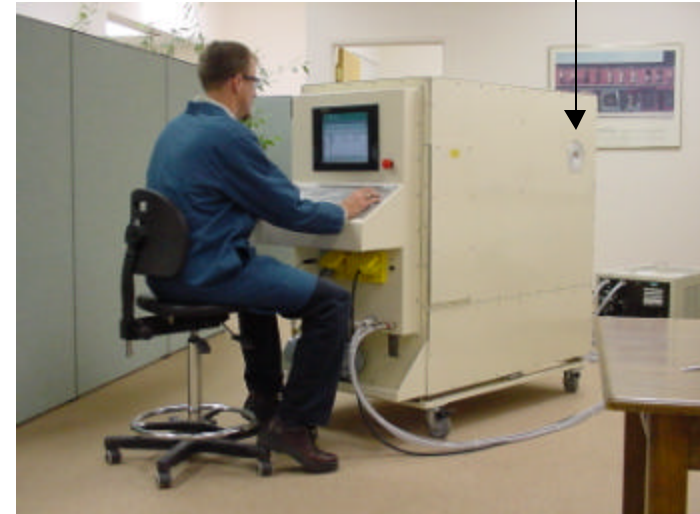
**Operating system**

# Sampling Process (CAM mode)

- System carousel is loaded with clean filters, blanks and spikes
- Carousel is loaded into system and sampling initiated
- Robot loads first filter into CAM port
- Air sample is obtained
- Robot moves filter to LIBS station for analysis
- Robot moves a new filter from the carousel to the CAM port
- Air sampling is initiated
- While the system obtaining another air sample, the robot moves back to the LIBS station and grabs the handle of the sample chamber
- Laser sparking and spectral detection sequence is initiated
- While sparking the robot moves the sample in multiple concentric orbits to spark the entire filter
- The spent filter is removed from chamber and disposed
- Spectral data evaluated with stored calibration algorithms
- Alarm initiated, if required
- Process repeated for entire filter carousel

# LIBS Spectra vs. Beryllium Mass



**Beryllium Peak (313 nm)**
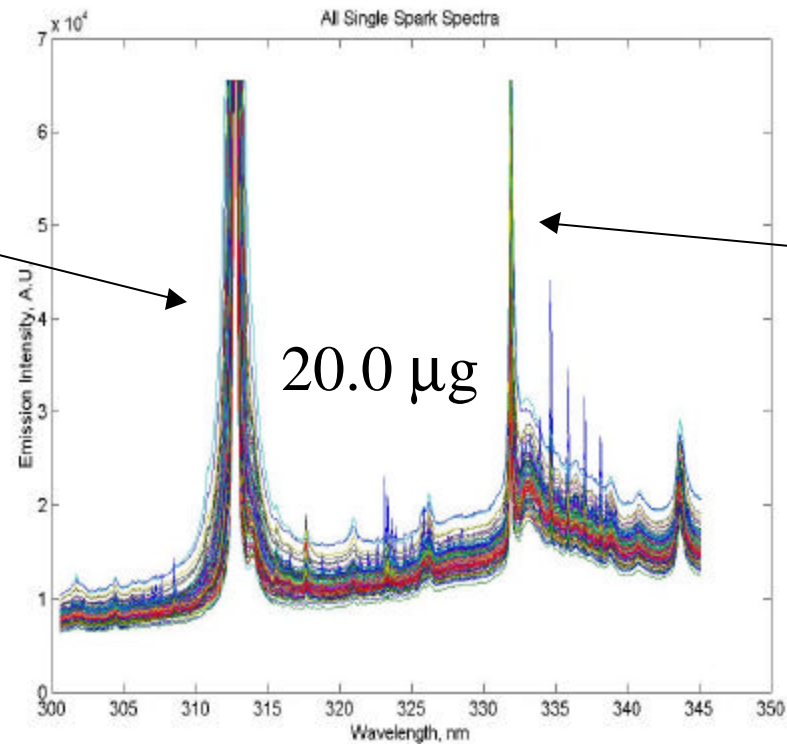
**Beryllium Peak (332 nm)**

20.0 µg

# Example LIBS Spectra of Be on Filters
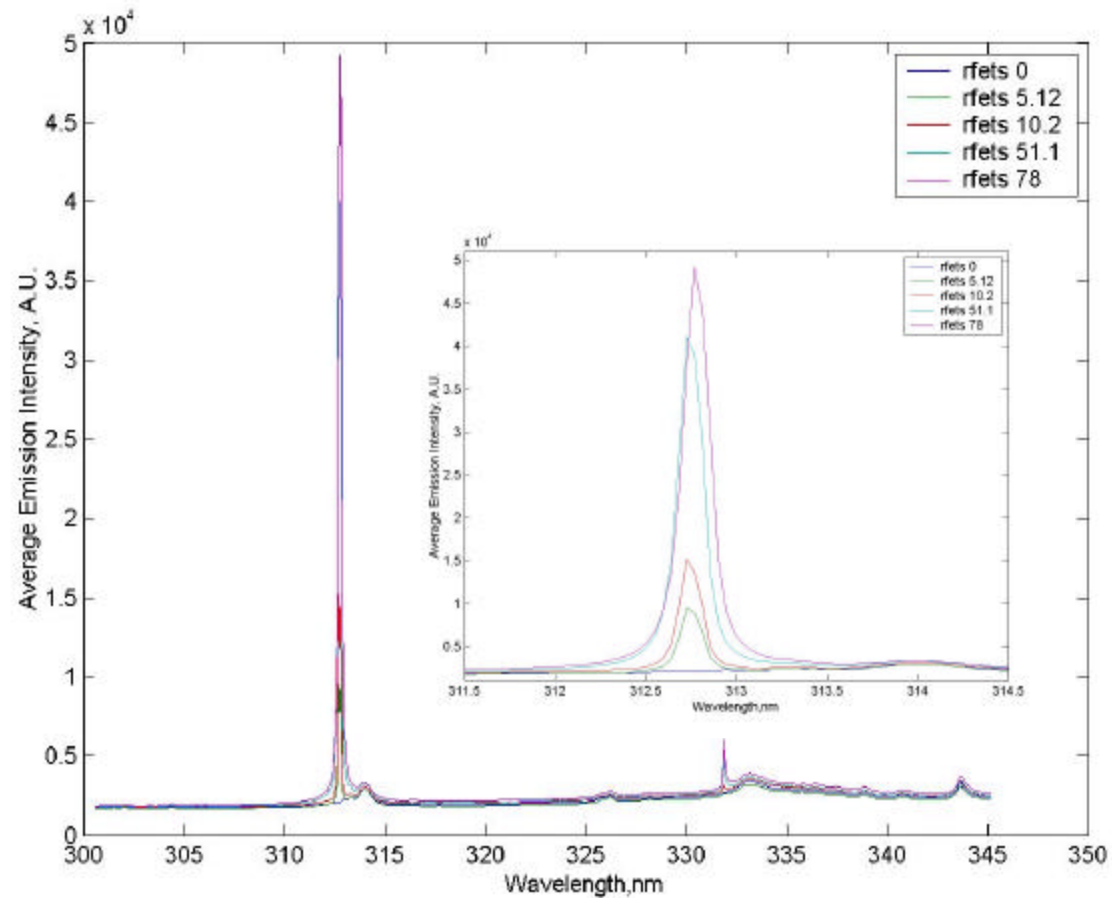
# LIBS Data Analysis

- Every spark for a filter is evaluated for data quality and beryllium mass
- Data Quality Acceptance – A primary and secondary data acceptance model is applied to each single-spark spectrum
  - Primary Data Acceptance – The purpose of this evaluation is to detect (and possibly diagnose) instrument malfunctions.
  - Secondary Data Acceptance – This evaluation detects radical changes in the chemical constituents in the sample matrix.
- Beryllium Mass Quantification – High/low concentrations
  - Detector Saturation – Each single-spark spectrum is evaluated for detector saturation and spectra are separated into two classes, those where saturation did not occur and those where it did
  - One of two models (Detector Saturated/Not Saturated) is applied to quantify the mass of Beryllium detected in a single spark.
- The total beryllium mass is the sum of the masses calculated for each of the sparks for a filter

# LIBS Data Classification Hierarchy
## Data Acceptance
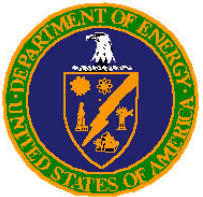
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                    ┌─────────────────────┐        ┌─────────────────┐
                    │  Raw Data Set       │        │ Primary Spectrum│
                    │  Multiple Spectra   │◄───────│   Acceptance    │
                    │  of Wavelength vs.  │        │     Model       │
                    │     Intensity       │        └─────────────────┘
                    └─────────┬───────────┘
            ┌─────────────────┴─────────────────┐
            ▼                                   ▼
  ┌──────────────────┐              ┌──────────────────┐      ┌─────────────┐
  │                  │              │                  │      │  Secondary  │
  │ Rejected Spectra │              │ Accepted Spectra │◄─────│  Spectrum   │
  │                  │              │                  │      │ Acceptance  │
  └──────────────────┘              └────────┬─────────┘      │    Model    │
                                             │                └─────────────┘
                               ┌─────────────┴─────────────┐
                               ▼                           ▼
                   ┌──────────────────┐          ┌──────────────────┐    ┌─────────────┐
                   │ Sample Spectra   │          │ Sample Spectra   │    │             │
                   │ Which Contain    │          │ Which Contain    │◄───│  Beryllium  │
                   │ Elements Not     │          │ Only Elements    │    │  Detection  │
                   │ Previously       │          │ Previously       │    │    Model    │
                   │ Detected         │          │ Detected         │    └─────────────┘
                   └──────────────────┘          └────────┬─────────┘
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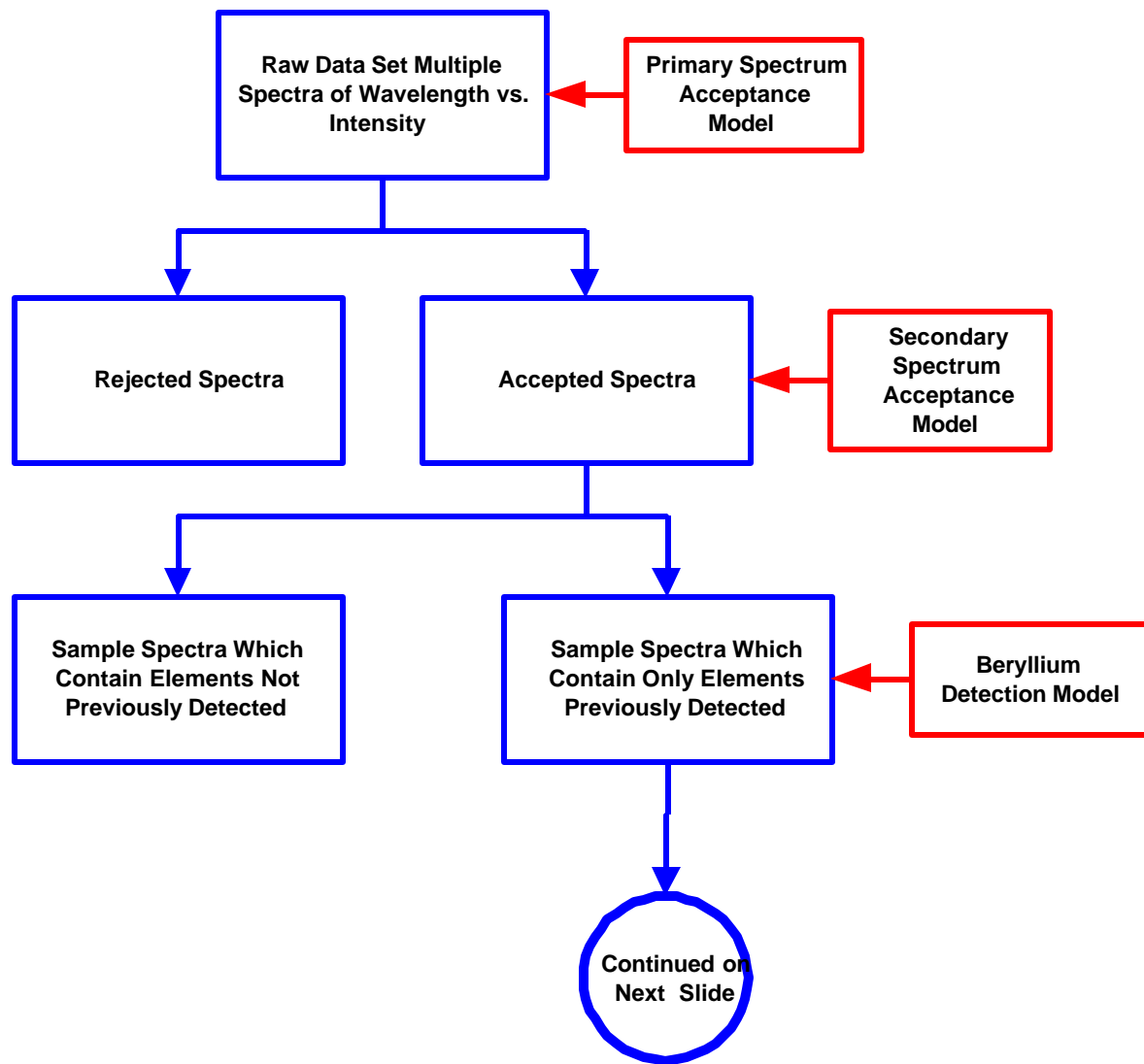
# LIBS Data Classification Hierarchy
## Beryllium Mass Quantification

**From Previous Slide**

**Spectra Where No Beryllium Was Detected**

**Spectra Where Beryllium Was Detected** ← **Detector Saturation Model**

**Spectra Where Detector Was Not Saturated on 313 nm Be Emission** ← **Primary Beryllium Mass Prediction Model**

**Spectra Where Detector Was Saturated on 313 nm Be Emission** ← **Secondary Beryllium Mass Prediction Model**

**Mass of Beryllium Detected**

**Mass of Beryllium Detected**

**Add up mass of Beryllium detected in each spark** → **Total Mass of Beryllium Detected on Filter**

# RFETS Ambient Dust
## LIBS Signal

# Scope of Work - Status

**WBS 1.0**
**Development of a Real-Time Measurement System for Airborne and Surfae Beryllium**

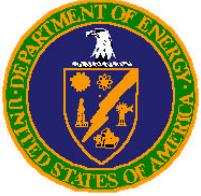| WBS 1.1 Project Planning | WBS 1.2 Instrument Design | WBS 1.3 System Fabrication | WBS 1.4 Instrument Calibration | WBS 1.5 Field Testing | WBS 1.6 System Delivery |
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**Completed**

# Schedule/Cost Status

- Schedule
  - System Design
    - System design completed on schedule
  - System Fabrication
    - All subsystems and components fabricated
    - System integration and testing ongoing
    - System fabrication schedule slightly delayed due to:
      - Laser performance issues (returned to supplier and fixed)
      - Sample chamber design issues (presently being modified)
    - Final system integration to be completed by early November
  - System Calibration
    - Calibration methodology completed
    - Final calibration awaiting final fabricated instrument
  - System Field Demonstration
    - RFETS testing scheduled late-November
    - Wipe and CAM testing
  - System Delivery – On schedule
- Cost – within budget

# Schedule
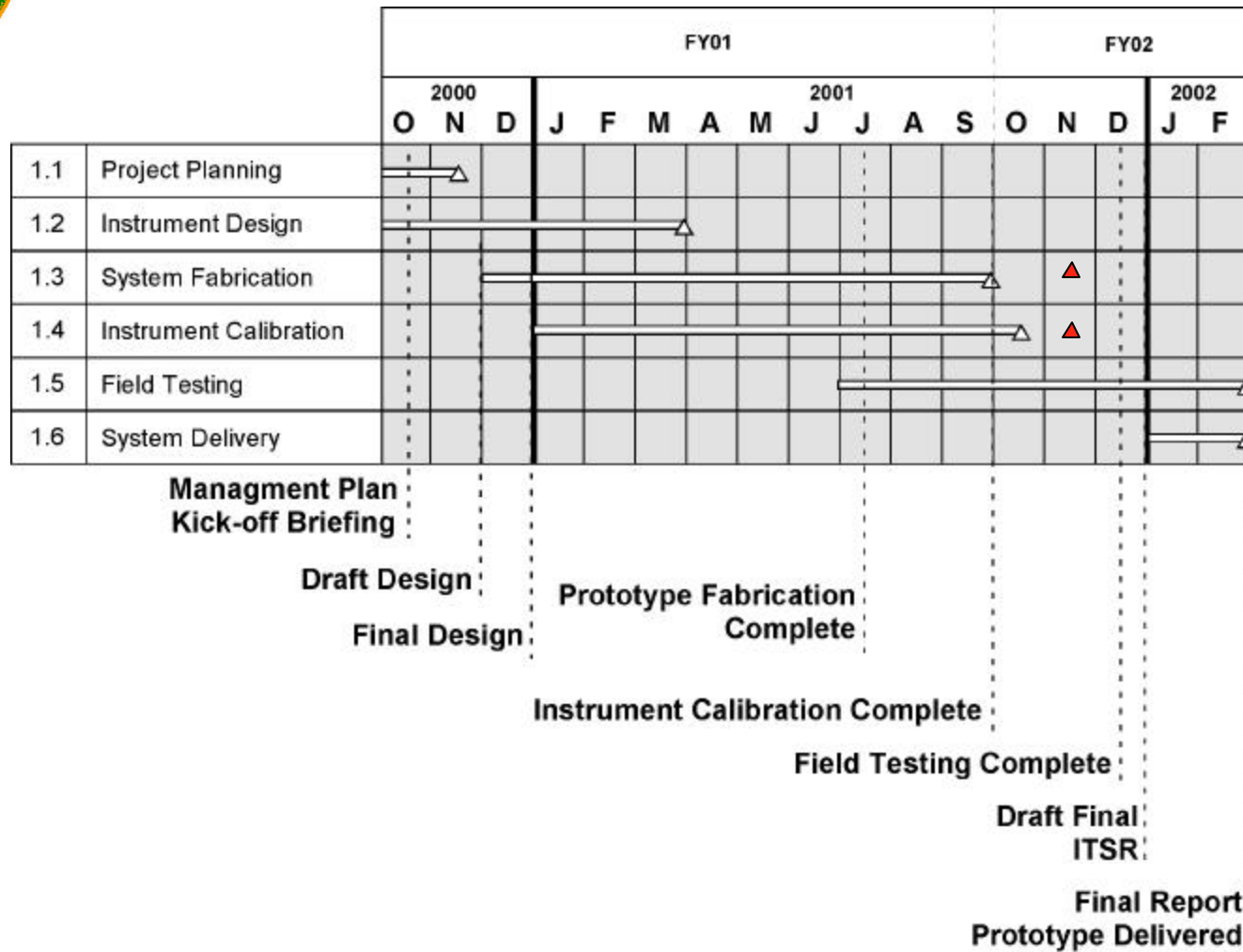
# Acknowledgements

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  - Dr. Adam Hutter
- **U.S. DOE Special Technology Laboratory**
  - Dr. Stephen Weeks
- **Lovelace Respiratory Research Institute**
- **National Institute for Occupational Health**
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